Conclusion

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► To cite this version:

C. Vettier, A. Wright. Conclusion. Revue de Physique Appliquée, 1984, 19 (9), pp.R9-R10.
10.1051/rphysap:019840019090R900 . jpa-00245277

HAL Id: jpa-00245277

<https://hal.science/jpa-00245277>

Submitted on 1 Jan 1984

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Conclusion

The workshop on « Sample Environments in X-ray and Neutron Experiments » is part of a series of joint meetings organized by European neutron research centres. After having discussed improvements of neutron and X-ray techniques, including sources and detection, it was felt quite important to discuss possibilities of better conditioning of samples under investigation. Indeed, only about 30 % of the neutron scattering research proposals submitted to ILL deal with experiments under ambient conditions. An increasing number of experiments require extreme conditions of temperature or pressure ; and 10 % of the proposals involve a magnetic field. Sample environment development must therefore logically keep pace with scattering techniques.

Such developments require collaborations between different groups of experts. During this workshop, we have tried to bring specialists in sample environment techniques together with neutron and X-ray scatterers in order to stimulate fruitful collaborations. Neutron and X-ray scattering were treated on the same basis because the availability of intense sources have opened up a new area for complementary studies. It would be impossible to review here all the topics which were developed during the sessions of the meeting. However a few major achievements should be pointed out as well as prospective guide lines for the near future.

In the field of high temperature, it is now evident that experiments at temperatures up to 2 000 °C are readily (if not always routinely) achievable at most major centres, and $T \geq 2\,500$ °C is a reasonable goal for the next few years. Beyond this, materials problems become very severe and each sample and mode of measurement must be specifically examined. Whilst considerable effort has been expended on achieving exotic temperatures, there is much room for improvement in the knowledge of the real sample temperature and the gradient across the sample. For modest temperatures, the use of multiple position temperature measurement is now replacing the single thermocouple of earlier furnaces and the use of a differential thermocouple is proposed for the precise measurement of gradients across the sample zone. The need for this temperature precision comes from the growing interest in precursors to phase-transitions ranging from critical phenomena to incommensurable phases, where a sample temperature variation of less than 1/1 000 °C over several days may be necessary for the experimental program.

At the very highest temperatures the prospect for precise temperature measurement is less clear. Accurate optical pyrometry remains a difficult and expensive technique which does not always match perfectly to the X-ray or neutron measurement, but is the only real alternative above 2 000 °C. Fortunately internal calibration from lattice parameter determinations can help where a well documented material such as tungsten is used as the sample container.

Finally the adaptation of high temperature equipment to the diffractometers or spectrometers is receiving more attention. The background scattering due to the sample environment can be very low indeed, and the loss of instrument measuring time due to setting up the equipment is a considered parameter of modern designs.

New flexible dilution refrigerators have been developed. They offer temperature in the 20 mK range. One important feature is that they can be used for different types of measurements, although they have been developed specifically for neutron scattering experiments. Ultra low temperature, in the mK region can also be achieved ; in neutron diffraction experiments, one major problem is the heating of nuclei by neutron capture.

These very low temperature devices can be used in a fixed scattering-plane geometry or normal beam geometry. At higher temperatures ($3 < T < 300$ K), there are now possibilities of rotating a single crystal sample inside the cryostat or using a four circle geometry. Much progress has been made on temperature control. Microprocessor systems can operate automatic temperature control in the range of 2 K to 2 000 K.

The use of time synchronized detection has already yielded considerable insight concerning biology and polymers physics essentially. Different types of problems can be investigated : rela-

xation from non equilibrium towards equilibrium; periodic perturbations from equilibrium states; steady non-equilibrium states. For example applied electrical field, mechanical stress can be cycled. Stopped-flow experiments can be readily adapted to a repetitive mode. Constant shear gradients are easily established in Couette-flow cell. However, a crucial parameter for the design of these experiments is the time required to install non-equilibrium conditions.

Studies of materials subjected to pressure can be performed by means of both X-ray and neutron scattering. However X-ray experiments allow for much higher attainable pressure than neutron, even though they are more absorbed. The reason for this is that X-rays require a smaller sample volume. Typically, X-ray experiments can be performed at pressure higher than 20 GPa whereas devices for neutron scattering can hold pressure up to 5 GPa. Improvements of apparatus are still desirable especially in the case of small angle neutron scattering which is of importance for molecular biology. In a near future, neutron diffraction experiments on single crystals subjected to 30 kbar will be possible on a 4-circle machine.

Finally, it is hoped that this workshop could stimulate new progress in Environment techniques which are vital for the successful conclusion of many X-ray and neutron scattering experiments.

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